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# मानक

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“Step Out From the Old to the New”

IS 3660-10 (2000): Methods of Test for Natural Rubber, Part 10: Determination of Nitrogen Content [NR:11] [PCD 13: Rubber and Rubber Products]



“ज्ञान से एक नये भारत का निर्माण”

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“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक  
प्राकृतिक रबड़ की परीक्षण विधियाँ  
भाग 10 नाइट्रोजन अंश ज्ञात करना  
[एन आर:11]  
( पहला पुनरीक्षण )

*Indian Standard*  
**METHODS OF TEST FOR NATURAL RUBBER**  
**PART 10 DETERMINATION OF NITROGEN CONTENT**  
**[NR:11]**  
( *First Revision* )

ICS 83.060

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Methods of test for natural rubber had been originally covered in the following four parts:

IS 3660 (Part 1) : 1972	Determination of dirt, volatile matter, ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disk viscometer), and mixing and vulcanizing of rubber in a standard compound [subsequently designated as IS 3660 (Part 51) : 1972]
IS 3660 (Part 2) : 1968	Determination of solvent extract and nitrogen content [subsequently designated as IS 3660 (Part 52) : 1980]
IS 3660 (Part 3) : 1971	Plasticity and plasticity retention index [subsequently withdrawn with the publication of Parts 11 and 12 of IS 3660 in 1989]
IS 3660 (Part 4) : 1979	Determination of colour, accelerated storage-hardening test and vulcanization characteristics (MOD test) [subsequently designated as IS 3660 (Part 54) : 1979]

While reviewing various test methods for natural rubber, the committee decided to align them with the corresponding international standards. Unification of test methods for natural and synthetic rubber has not been considered necessary. However, in revising test methods for natural rubber, the committee had decided to revise and split the standards into further parts and publish test methods for individual characteristics under natural rubber (NR) series. For proper referencing of the original test methods and the new methods revised/ under revision, a table showing correspondence of the various methods of test covered in the previous 4 parts of IS 3660, namely (Parts 1, 2, 3 and 4) with the presently split parts have been given in Annex A. In order to facilitate cross reference, it has been decided to retain the original discrete NR series numbers assigned to various test methods in original IS 3660 (Parts 1, 2, 3 and 4) in the revised part of IS 3660.

The test method given in this revised standard will supersede the test method as given under NR : 11 of IS 3660 (Part 52) : 1968. This standard (Part 52) shall be withdrawn upon its complete revision.

In the preparation of this standard, assistance has been derived from ISO 1656 : 1996 'Rubber, raw natural and natural latex, natural—Determination of nitrogen content', published by the International Organization for Standardization (ISO).

In this revision Macro method has been incorporated and Semi-micro method has been modified. In addition, modifications necessary to align it with latest ISO standard, namely, ISO 1656 : 1996.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

# Indian Standard

## METHODS OF TEST FOR NATURAL RUBBER

### PART 10 DETERMINATION OF NITROGEN CONTENT

[NR:11]

( *First Revision* )

#### 1 SCOPE

This Indian Standard prescribes a Macro method and a Semi-micro method for the determination of nitrogen in raw natural rubber and in natural rubber latex using variants of the Kjeldahl process.

NOTE — The determination of nitrogen in natural rubber is usually carried out in order to arrive at an estimate of the protein content. Minor amounts of non-proteinous nitrogen containing constituents are also present, however, and in the dry solids prepared from natural rubber latex these materials can make a substantial contribution to the total nitrogen content.

#### 2 NORMATIVE REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
266 : 1993	Sulphuric acid ( <i>third revision</i> )
1070 : 1992	Reagent grade water ( <i>third revision</i> )
3660	Methods of test for natural rubber:
(Part 8) : 1999	Part 8 Mixing and vulcanizing of rubber in standard compound (NR:9) ( <i>second revision</i> )
(Part 11) : 1989	Part 11 Determination of plasticity (NR:12) ( <i>first revision</i> )
5599 : 1999	Rubber—Raw, natural and synthetic — Methods of sampling and sample preparation ( <i>first revision</i> )

#### 3 PRINCIPLE

A known mass of the sample is digested with a mixture of sulphuric acid, potassium sulphate and catalytic amounts of copper sulphate and selenium or sodium selenate, thereby converting nitrogen compounds into ammonium hydrogen sulphate from which the ammonia is distilled after making the mixture alkaline.

The distilled ammonia is absorbed:

- a) either in standard volumetric sulphuric acid solution followed by titration of the excess acid with a standard volumetric base solution;
- b) or in boric acid solution followed by titration

with standard volumetric acid solution (as boric acid is a weak acid, it does not affect the indicator used for this titration).

#### 4 MACRO METHOD

##### 4.1 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity conforming to IS 1070.

##### 4.1.1 Catalyst Mixture or Catalyst Solution

CAUTION — When working with selenium, avoid breathing powder and/or contact with skin or clothing. Work only with adequate ventilation.

##### 4.1.1.1 Catalyst mixture

Prepare a finely divided intimate mixture of

- 30 parts by mass of anhydrous potassium sulphate ( $K_2SO_4$ );
- 4 parts by mass of copper sulphate pentahydrate ( $CuSO_4 \cdot 5H_2O$ ); and
- 1 part by mass of selenium powder, or 2 parts by mass of sodium selenate decahydrate ( $Na_2SeO_4 \cdot 10H_2O$ ).

##### 4.1.1.2 Catalyst solution

Dissolve, with heating

- 110 g of anhydrous potassium sulphate,
- 14.7 g of copper sulphate pentahydrate, and
- 3.7 g of selenium, or 7.49 g of sodium selenate, in 600 ml of sulphuric acid (4.1.2).

##### 4.1.2 Sulphuric Acid

Conforming to IS 266.

##### 4.1.3 Sulphuric Acid

Standard volumetric solution,  $c(H_2SO_4) = 0.05$  mol/l 000 ml.

##### 4.1.4 Sodium Hydroxide

Standard volumetric solution,  $c(NaOH) = 0.1$  mol/l 000 ml.

##### 4.1.5 Sodium Hydroxide Solution

$c(NaOH) = 17$  mol/l 000 ml.

Dissolve 680 g of solid sodium hydroxide in water and make up to 1 000 ml.

#### 4.1.6 Boric Acid Solution

Dissolve 20 g of solid boric acid in 1 000 ml of water, warming if necessary, and cool the solution to room temperature.

#### 4.1.7 Mixed Indicator Solution

Dissolve 0.1 g of methyl red and 0.05 g of methylene blue in 100 ml of 96 percent (v/v) ethanol.

This indicator may deteriorate during storage and shall therefore be freshly prepared.

### 4.2 Apparatus

Ordinary laboratory apparatus and Kjeldahl apparatus, with a digestion flask of 800 ml capacity.

### 4.3 Sampling and Preparation of Test Portion

**4.3.1** For the determination of nitrogen in raw solid rubber, a test portion shall be taken from the homogenized piece.

**4.3.2** For the determination of nitrogen in latex, a representative portion of thoroughly mixed latex containing about 2 g of total solids shall be dried to constant mass.

### 4.4 Procedure

**4.4.1** Cut about 2 g of the rubber or dried latex, weighed to the nearest 0.5 mg, into small pieces and place in the digestion flask (see 4.2). Add about 13 g of the catalyst mixture (4.1.1.1) and 60 ml of the sulphuric acid (4.1.1.2) or, alternatively, 65 ml of the catalyst solution (4.1.1.2). Mix the contents of the flask by swirling, and then boil gently until the solution is clear. Continue boiling for a further period of 60 min. Allow the digestion flask and its contents to cool to room temperature, cautiously add 200 ml of water and mix by swirling. Place the receiving flask containing the absorbing solution in position, connect the distillation unit, then slowly add 150 ml of the sodium hydroxide solution (4.1.5) to the digestion flask from a dropping funnel.

**4.4.2** Carry out the absorption and titration of the liberated ammonia by the procedure described in 4.4.2.1 or 4.4.2.2. The temperature of the receiving flask shall be maintained below 30°C to prevent any loss of ammonia.

**4.4.2.1** Place 75 ml of water and, by means of a pipette, 25 ml of the standard volumetric sulphuric acid solution (4.1.3) in the receiving flask of the distillation unit, together with two drops of mixed indicator solution (4.1.7). Position the receiving flask so that the end of the delivery tube from the condenser dips below the surface of the absorbing solution. While

holding the stopper of the digestion flask in place, thoroughly mix the contents by swirling. Immediately commence distillation and continue at a steady rate until 200 ml of distillate have been collected. If the colour of the indicator changes, indicating alkalinity of the absorbing solution, discontinue the determination and repeat the procedure using more sulphuric acid or a smaller test portion. When the distillation is complete (normally when the volume in the flask reaches about 300 ml), titrate the contents with the sodium hydroxide solution (4.1.4), reading the burette to the nearest 0.05 ml.

**4.4.2.2** Place 200 ml of the boric acid solution (4.1.6) in the receiving flask of the distillation unit with two drops of the mixed indicator solution (4.1.7). Carry out the distillation as described in 4.4.2.1 and titrate the distillate with the sulphuric acid solution (4.1.3), reading the burette to the nearest 0.05 ml.

### 4.5 Blank Test

In parallel with the determination, carry out a blank test with the same quantities of reagents under the same operating conditions, but omitting the test portion.

### 4.6 Expression of Results

**4.6.1** When sulphuric acid is used as the absorbing solution as specified in 4.4.2.1, the nitrogen content of the rubber, expressed as a percentage by mass, is given by the formula:

$$\text{Nitrogen content, percent by mass} = \frac{0.14 (V_2 - V_1)}{m}$$

where

$V_1$  = volume, in ml, of sodium hydroxide solution (4.1.4) required for the titration;

$V_2$  = volume, in ml of sodium hydroxide solution (4.1.4) required for the titration in the blank test; and

$m$  = mass, in grams, of the test portion.

Express the result to the nearest 0.01 percent.

**4.6.2** When boric acid is used as the absorbing solution as specified in 4.4.2.2, the nitrogen content of the rubber, expressed as a percentage by mass, is given by the formula:

$$\text{Nitrogen content, percent by mass} = \frac{0.14 (V_3 - V_4)}{m}$$

where

$V_3$  = volume, in ml, of sulphuric acid solution (4.1.3) required for the titration;

$V_4$  = volume, in ml of sulphuric acid solution (4.1.3) required for the titration in the blank test; and

$m$  = mass, in grams, of the test portion.

Express the result to the nearest 0.01 percent.

## 5 SEMI-MICRO METHOD

### 5.1 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

#### 5.1.1 Catalyst Mixture

**CAUTION** — When working with selenium, avoid breathing vapours and/or contact with skin or clothing. Work only with adequate ventilation.

Prepare a finely divided intimate mixture of

- 30 parts by mass of anhydrous potassium sulphate ( $K_2SO_4$ );
- 4 parts by mass of copper sulphate pentahydrate ( $CuSO_4 \cdot 5H_2O$ ); and
- 1 part by mass of selenium powder, or 2 parts by mass of sodium selenate decahydrate ( $Na_2SeO_4 \cdot 10H_2O$ ).

#### 5.1.2 Sulphuric Acid

Conforming to IS 266.

#### 5.1.3 Sulphuric Acid

Standard volumetric solution,  $c(H_2SO_4) = 0.005$  mol/1 000 ml.

#### 5.1.4 Sodium Hydroxide

Solution  $c(NaOH) = 10$  mol/1 000 ml.

Dissolve 400 g of solid sodium hydroxide in 600 ml of water.

#### 5.1.5 Sodium Hydroxide

Standard volumetric solution,  $c(NaOH) = 0.01$  mol/1 000 ml carbonate-free.

#### 5.1.6 Boric Acid

Solution  $c(H_3BO_3) = 0.32$  mol/1 000 ml.

Dissolve 20 g of solid boric acid in 1 litre of water, warming if necessary, and cool the solution to room temperature.

#### 5.1.7 Mixed Indicator Solution

Dissolve 0.1 g of methyl red and 0.05 g of methylene blue in 100 ml of 95 percent (v/v) ethanol.

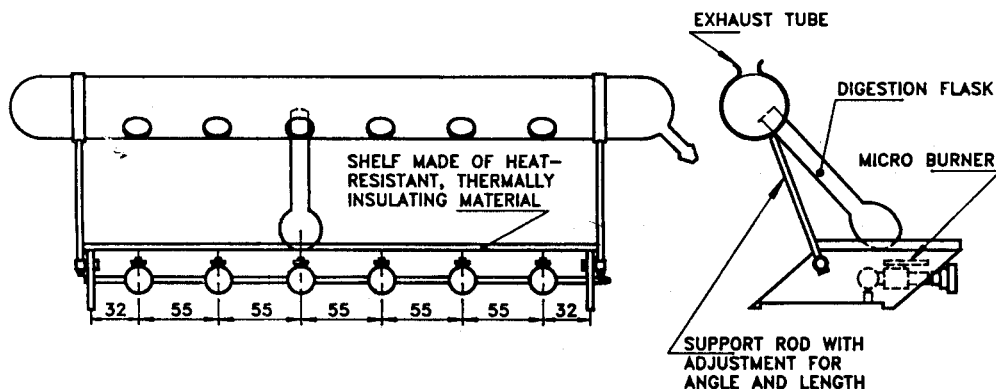
This indicator may deteriorate during storage and shall therefore be freshly prepared.

### 5.2 Apparatus

Ordinary laboratory apparatus.

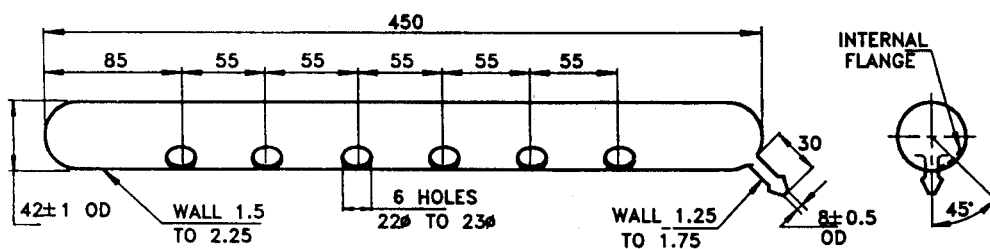
#### 5.2.1 Semi-Micro Kjeldahl Digestion Apparatus

With digestion flasks of 30 ml (see Fig. 1, 2 and 3).



All dimensions in millimetres.

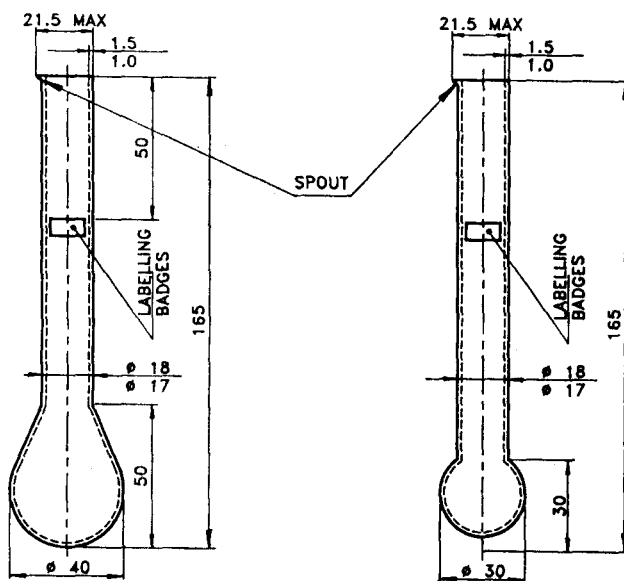
FIG. 1 ASSEMBLY OF DIGESTION APPARATUS FOR THE SEMI-MICRO METHOD



All dimensions in millimetres.

FIG. 2 EXHAUST TUBE FOR THE SEMI-MICRO METHOD





**All dimensions in millimetres.**

FIG. 3 DIGESTION FLASKS FOR THE SEMI-MICRO METHOD

### 5.2.2 Semi-Micro Kjeldahl Distillation Unit

With a condenser tube of silver, borosilicate glass or tin (see Fig. 4 to 9).

### 5.2.3 Semi-Micro Burette

Capacity 10 ml, graduated in 0.02 ml divisions.

### 5.3 Sampling and Preparation of Test Portion

For the determination of nitrogen in raw solid rubber, a test portion shall be taken from the homogenized piece.

For the determination of nitrogen in latex, a representative portion of thoroughly mixed latex containing about 0.1 g of total solids shall be dried to constant mass.

## 5.4 Procedure

**5.4.1** Weigh, to the nearest 0.1 mg, 0.1 to 0.2 g of the rubber or dried latex and place in digestion flask (*see* 5.2.1). Add about 0.65 g of the catalyst mixture (5.1.1) and 3.0 ml of the sulphuric acid (5.1.2) and heat the contents carefully to the boiling point. Continue boiling for a further 30 min after the digest has become clear and green with no yellow tint. Avoid excess boiling, as indicated by a tendency for the digest to solidify on cooling, since this may lead to loss of nitrogen. Bring the water in the steam generator of the distillation unit to the boil and pass steam through the distillation apparatus (5.2.2), including the receiving flask for at least 2 min. The water-jacket of the condenser shall be empty of water during the steaming-out operation. Meanwhile, cool the digestion flask to room temperature or below, add 10 ml of water

and immediately transfer the contents to the distillation flask at the conclusion of the steaming-out process. Complete the transfer by rinsing three times with 3 ml portions of water and draining the flask thoroughly after each transfer.

**5.4.2** Discard any condensate which has collected in the receiver and complete the distillation and titration of ammonia by the procedure described in 5.4.2.1 or 5.4.2.2. The temperature of the receiving flask shall be maintained below 30°C to prevent loss of ammonia.

**5.4.2.1** Add from the semi-micro burette (5.2.3) to the steamed-out receiver of the distillation apparatus a measured volume of sulphuric acid solution (5.1.3), using at least 10 ml (the exact volume depending on the amount of nitrogen expected), together with two drops of the mixed indicator solution (5.1.7) and about 10 ml of water. Position the receiver so that the end of the delivery tube from the condenser dips below the surface of the acid. It is an advantage to tilt the receiver slightly to gain a greater depth of liquid. Add approximately 10 ml of the sodium hydroxide solution (5.1.4) to the distillation flask by means of a measuring cylinder, and pass steam from the generator through the distillation flask for 10 to 12 min at such a rate that the final volume of liquid in the receiver is about 50 ml. If the colour of the indicator changes, indicating alkalinity of the absorbing solution, discontinue the determination and repeat the procedure using more sulphuric acid or a smaller test portion. When the distillation is complete, lower the receiving flask until the tip of the condenser is above the level of the acid, continue the distillation for another 1 min, and then rinse the tip of the condenser tube with a few millilitres

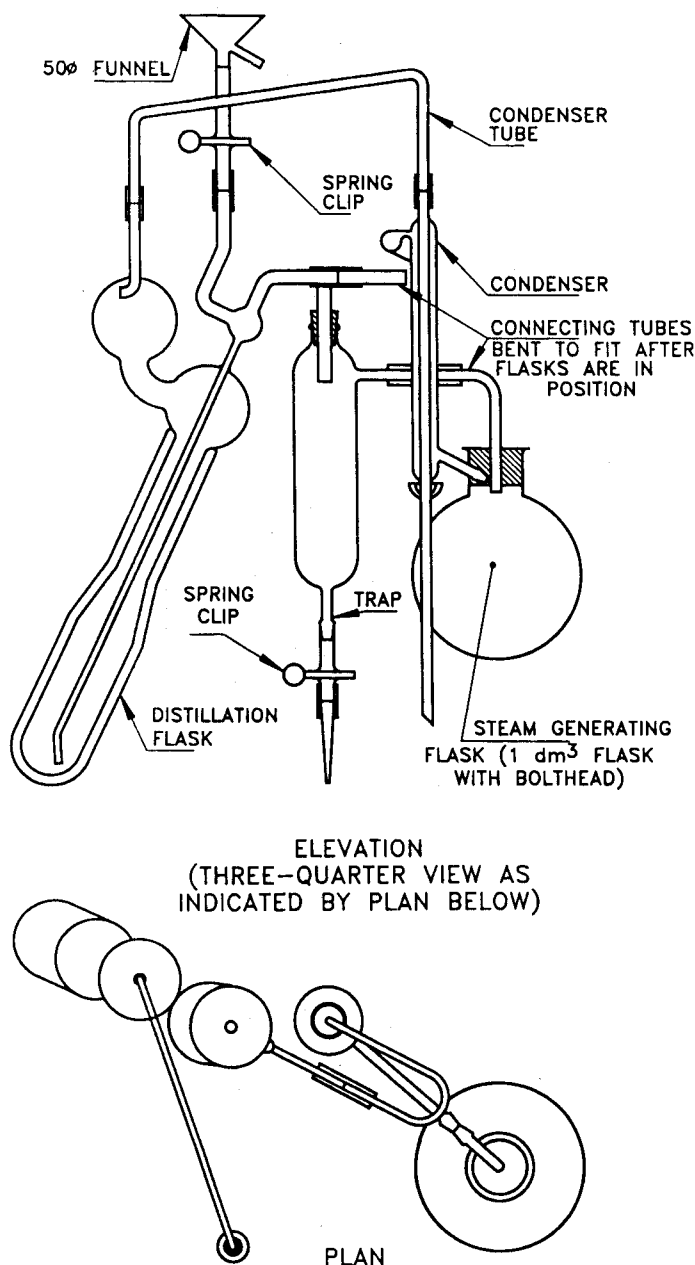


FIG. 4 ASSEMBLY OF DISTILLATION APPARATUS FOR THE SEMI-MICRO METHOD

of water which shall be collected in the distillate. Immediately titrate the contents of the receiving flask with the sodium hydroxide solution (5.1.5), reading the burette to the nearest 0.02 ml.

**5.4.2.2** Place about 10 ml of the boric acid solution (5.1.6) in the steamed-out receiving flask with two drops of the mixed indicator solution (5.1.7) and add 10 ml of distilled water. Carry out the distillation as described in 5.4.2.1, but note that, in the presence of boric acid, the indicator colour should change immediately distillation of ammonia commences. Titrate the distillate with sulphuric acid solution (5.1.3),

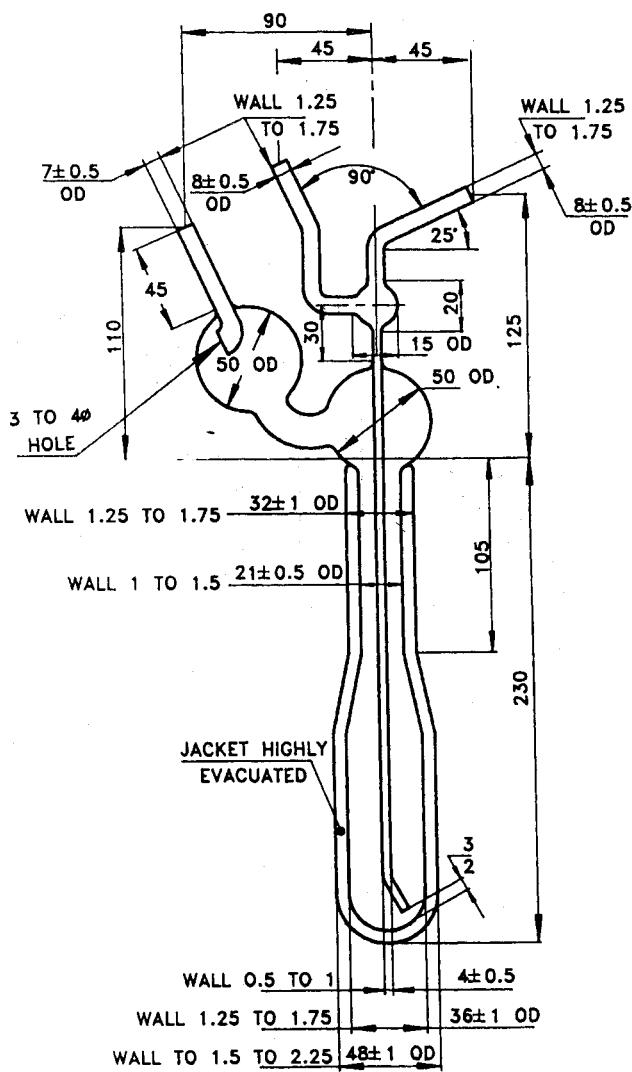
reading the burette to the nearest 0.02 ml.

### 5.5 Blank Test

In parallel with the determination carry out a blank test using the same quantities of reagents under the same operating conditions, but omitting the test portion.

### 5.6 Expression of Results

**5.6.1** When sulphuric acid is used as the absorbing solution as specified in 5.4.2.1, the nitrogen content of the rubber, expressed as a percentage by mass, is



All dimensions in millimetres.

FIG. 5 DISTILLATION FLASK FOR THE SEMI-MICRO METHOD

given by the formula below:

Nitrogen content,  
percent by mass =  $\frac{0.14 (V_2 - V_1)}{m}$

where

- $V_1$  = volume, in ml, of sodium hydroxide solution (5.1.5) required for the titration;
- $V_2$  = volume, in ml, of sodium hydroxide solution (5.1.5) required for the titration in the blank test; and

$m$  = mass, in grams, of the test portion.

Express the result to the nearest 0.01 percent.

5.6.2 When boric acid is used as the absorbing solution as specified in 5.4.2.2, the nitrogen content of the rubber, expressed as a percentage by mass, is given by the formula below:

Nitrogen content,  
percent by mass =  $\frac{0.114 (V_3 - V_4)}{m}$

where

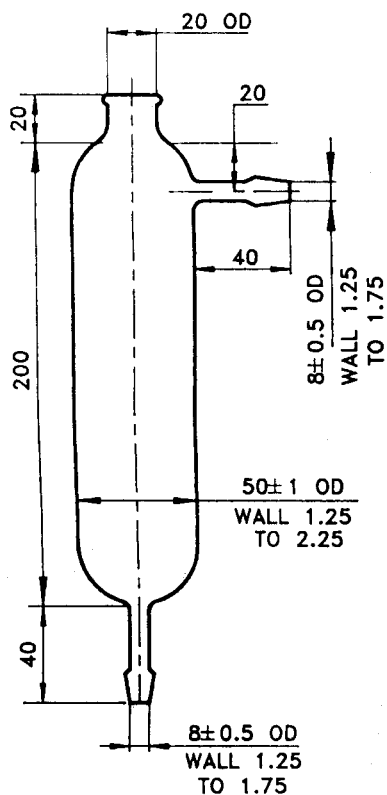
- $V_3$  = volume, in ml, of sulphuric acid solution (5.1.3) required for the titration;
- $V_4$  = volume, in ml, of sulphuric acid solution (5.1.3) required for the titration in the blank test; and
- $m$  = mass, in grams, of the test portion.

Express the result to the nearest 0.01 percent.

6 PRECISION

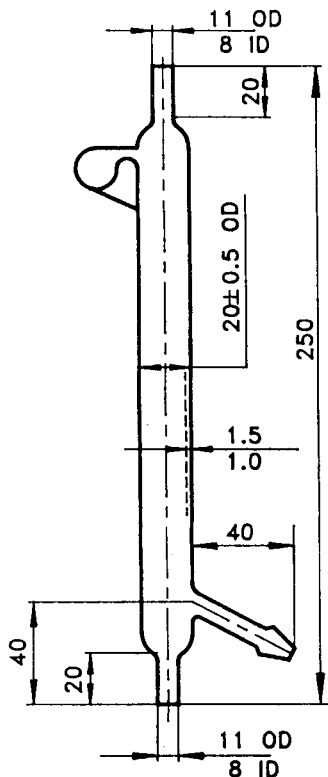
6.1 The precision calculations express repeatability and reproducibility.

6.2 For both the blended and the unblended samples, a test result was taken as the mean of three separate determinations.



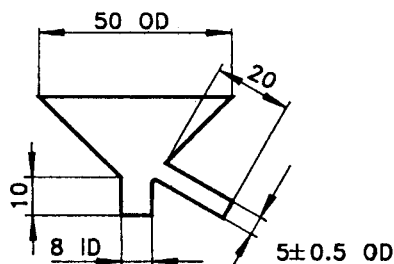
All dimensions in millimetres.

FIG. 6 TRAP FOR THE SEMI-MICRO METHOD



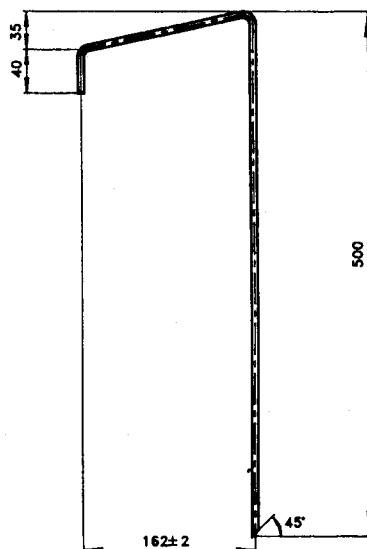
All dimensions in millimetres.

FIG. 7 CONDENSER JACKET FOR THE SEMI-MICRO METHOD



All dimensions in millimetres.

FIG. 8 DRIP FUNNEL FOR THE SEMI-MICRO METHOD



All dimensions in millimetres.

FIG. 9 CONDENSER TUBE FOR THE SEMI-MICRO METHOD

### 6.3 Precision Results

The precision results for the blended sample programme are given in Table 1 and the results for the unblended sample programme in Table 2.

### 7 TEST REPORT

The test report shall include the following information:

- A reference to this standard and the method used;
- All details necessary for the identification of the material tested;
- The results and the units in which they are expressed;
- Any unusual features noted during the determination;
- Any operation not included in this standard or in the standards to which reference is made, and any operation regarded as optional; and
- The date of the test.

Table 1 Type 1 Precision — Blended Sample Testing  
(Clause 6.3)

Rubber Sample	Average Nitrogen Content, Percent, m/m	Within-Laboratory Repeatability		Inter-Laboratory Reproducibility	
		<i>r</i>	( <i>r</i> )	<i>R</i>	( <i>R</i> )
(1)	(2)	(3)	(4)	(5)	(6)
A	0.45	0.053	11.7	0.094	20.9
B	0.53	0.024	4.45	0.161	30.2
Pooled values	0.49	0.042	8.42	0.127	25.6
<i>r</i> = repeatability, percent by mass;					
( <i>r</i> ) = repeatability, percent (relative) of the average;					
<i>R</i> = reproducibility, percent by mass; and					
( <i>R</i> ) = reproducibility, percent (relative) of the average.					

Table 2 Type 1 Precision — Unblended Sample Testing  
(Clause 6.3)

Rubber Sample	Average Nitrogen Content, Percent, m/m	Within-Laboratory Repeatability		Inter-Laboratory Reproducibility	
		<i>r</i>	( <i>r</i> )	<i>R</i>	( <i>R</i> )
(1)	(2)	(3)	(4)	(5)	(6)
A	0.36	0.021	5.83	0.189	52.7
B	0.36	0.038	10.82	0.185	51.9
Pooled values	0.36	0.031	8.67	0.187	52.4
See Table 1 for symbol definitions.					

ANNEX A  
(Foreword)

TABLE SHOWING CORRESPONDENCE OF THE VARIOUS METHODS OF TESTS COVERED  
IN THE ORIGINAL IS 3660 (PART 1) : 1972 (NOW DESIGNATED AS PART 51),  
IS 3660 (PART 2):1968 (NOW DESIGNATED AS PART 52), IS 3660 (PART 3) : 1971  
(SINCE WITHDRAWN ON ITS COMPLETE REVISION), AND IS 3660 (PART 4) : 1979  
(NOW DESIGNATED AS PART 54) WITH THE NEW REVISED STANDARDS

Test Method	IS No.	Original Part (Series)	IS No.	New Parts Series	Remarks/ Reaffirmed
(1)	(2)	(3)	(4)	(5)	(6)
NR Series					
Determination of dirt	3660 : 1972	Part 1 (NR:1)	3660 (Part 1) : 1999	(NR:1)	—
Determination of volatile matter	3660 : 1972	Part 1 (NR:2)	3660 (Part 2) : 1985	(NR:2)	Dec 1995
Determination of ash	3660 : 1972	Part 1 (NR:3)	3660 (Part 3) : 1988	(NR:3)	Jan 1995
Determination of total copper	3660 : 1972	Part 1 (NR:4)	3660 (Part 4) : 1988	(NR:4)	Jan 1995
Determination of manganese	3660 : 1972	Part 1 (NR:5)	3660 (Part 5) : 1989	(NR:5)	Jan 1995
Determination of iron	3660 : 1972	Part 1 (NR:6)	Deleted since this test is no longer being done		
Determination of rubber hydrocarbon	3660 : 1972	Part 1 (NR:7)	3660 (Part 6) : 1988	(NR:7)	Jan 1995
Determination of vis-	3660 : 1972	Part 1 (NR:8)	3660 (Part 7) : 1988	(NR:8)	Jan 1995

<i>Test Method</i>	<i>IS No.</i>	<i>Original Part (Series)</i>	<i>IS No.</i>	<i>New Parts Series</i>	<i>Remarks/ Reaffirmed</i>
(1)	(2)	(3)	(4)	(5)	(6)
Determination of viscosity by shearing disk viscometer	3660 : 1972	Part 1 (NR:8)	3660 (Part 7) : 1988	(NR:8)	Jan 1995
Mixing and vulcanizing of rubber in a standard compound	3660 : 1972	Part 1 (NR:9)	3660 (Part 8) : 1999	(NR:8)	—
Determination of solvent extract	3660 : 1968	Part 2 (NR:10)	3660 (Part 9) : 1989	(NR:10)	Jan 1995
Determination of nitrogen	3660 : 1968	Part 2 (NR:11)	3660 (Part 10) : 2000	(NR:11)	—
Determination of plasticity	3660 : 1971	Part 3 (NR:12)	3660 (Part 11) : 1989	(NR:12)	Jan 1995
Determination of plasticity retention index (PRI)	3660 : 1972	Part 3 (NR:13)	3660 (Part 12) : 1989	(NR:13)	Jan 1995
Determination of colour	3660 : 1979	Part 4 (NR:14)	3660 (Part 13) : 1997	(NR:14)	—
Determination of storage hardening test	3660 : 1979	Part 4 (NR:15)	3660 (Part 14)	(NR:15)	Revision still to be published
Determination of vulcanization characteristics (MOD test)	3660 : 1979	Part 4 (NR:16)	3660 (Part 15)	(NR:16)	do
Method of preparation of test samples	3660 : 1972	Part 1 (Clause 3)	included in the first revision of IS 5599 : 1999		

## NOTES

1 Original IS 3660 (Part 1) : 1972 now re-designated as IS 3660 (Part 51) : 1972.

2 Original IS 3660 (Part 2) : 1968 now re-designated as IS 3660 (Part 52) : 1968.

3 Subsequent to its complete revision, IS 3660 (Part 3) : 1971 has since been withdrawn.

4 Original IS 3660 (Part 4) : 1979 now re-designated as IS 3660 (Part 54) : 1979.

**Bureau of Indian Standards**

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**BUREAU OF INDIAN STANDARDS**

**Headquarters:**

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002  
Telephones: 323 01 31, 323 3375, 323 94 02

Telegrams: Manaksanstha  
(Common to all offices)

**Regional Offices:**

Telephone

Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	323 76 17, 323 38 41
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi CALCUTTA 700054	{ 337 84 99, 337 85 61 337 86 26, 337 91 20
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Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	{ 235 02 16, 235 04 42 235 15 19, 235 23 15
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